
पेट्रोलियम और उसके उत्पादों
के लिए परीक्षण के तरीके

[पी : 134]

राँ और कैल्सिंड पेट्रोलियम
कोक में वाष्पशील पदार्थ के निर्धारण
(पहला पुनरीक्षण)

**Methods of Test for
Petroleum and its Products**

[P : 134]

**Determination of Volatile Matter
in Raw and Calcined Petroleum Coke**
(*First Revision*)

ICS 75.160.30

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FOREWORD

This Indian Standard [P : 134] (First Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Methods of Sampling and Test for Petroleum, Petroleum Products, Gaseous Fuels and Lubricants Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Volatile matter in coke refers to the components of coke, except for moisture, which are liberated at high temperature in the absence of air. This is usually a mixture of short and long chain hydrocarbons, aromatic hydrocarbons and some sulfur. The volatile matter of coke is determined under controlled heating to $950 \pm 15^{\circ}\text{C}$. This test method is not satisfactory for deducting material content.

Volatile matter, when determined as herein described, can be used to establish the rank of coals, to indicate coke yield on carbonization process, to provide the basis for purchasing and selling, or to establish burning characteristics.

This standard was first published in 1989. In the first revision of this standard terminology has been included and scope has been redefined. This method is applicable to both coal and coke.

This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Specific precautionary statements are given in Section 8.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 134]

DETERMINATION OF VOLATILE MATTER IN RAW AND CALCINED PETROLEUM COKE

(*First Revision*)

1 SCOPE

1.1 This standard [P : 134] covers the test method for determination of the percentage of gaseous products, exclusive of moisture vapor, in the analysis sample which are released under the specific conditions of the test. results.

1.2 This test method for determination of volatile matter is empirical; because of its empirical nature, strict adherence to basic principles and permissible procedures is required to obtain valid

2 REFERENCES

The standards listed below contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication the editions indicated were valid. All standards are subject to revision, and parties to agreements based on the standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>Indian standard</i>	<i>Title</i>
1447 (Part 4) : 1989	Sampling of petroleum coke for laboratory analysis

3 TERMINOLOGY

3.1 Sparking Fuels

Within the context of this test method, fuels that do not yield a coherent cake as residue in the volatile matter determination but do evolve gaseous products at a rate sufficient to mechanically carry solid particles out of the crucible when heated at the standard rate. Such coals normally include all low-rank non caking coals and lignite's but can also include those anthracites, semi anthracites, bituminous and cokes that lose solid particles as described above. These are defined as sparking fuels because particles escaping at the higher temperatures can become incandescent and spark as they are emitted.

4 SUMMARY OF TEST METHOD

4.1 Volatile matter is determined by establishing the loss in weight resulting from heating a coal or coke under rigidly controlled conditions. The measured weight loss, corrected for moisture as determined in test method, establishes the volatile matter content. Two procedures are described to permit conformity with differences in sample behavior.

4.2 In this empirical test method, the use of platinum crucibles shall be considered the standard reference method for volatile matter. Platinum crucibles shall be used in determining the volatile matter of coke and volatile matter determined for classification of coals by rank. Volatile matter determinations by some laboratories using alternate nickel-chromium alloy crucibles having the physical dimensions specified in **5.3** have been shown to differ from those obtained using platinum crucibles. A laboratory utilizing nickel-chromium crucibles shall first determine if a relative bias exists between the use of nickel-chromium and platinum crucibles on the coals being tested using the test method set. Where a relative bias is shown to exist, the volatile matter determined using nickel-chromium crucibles shall be corrected by a factor determined through comparison of volatile matter results from both crucible types on coals being tested or analysis of samples of known proximate analysis

5 APPARATUS

5.1 Platinum Crucible

With closely fitting cover, for coal. The crucible shall be of not less than 10 or more than 20 ml capacity, not less than 25 or more than 35 mm in diameter, and not less than 30 or more than 35 mm in height.

5.2 Platinum Crucible

With closely fitting cover, for coke. The crucible shall be of 10 ml capacity, with capsule cover having thin flexible sides fitting down into crucible. Or the double-crucible method can be used, in which the sample is placed in 10 ml platinum crucible, which is then

covered with another crucible of such a size that it will fit closely to the sides of the outer crucible and its bottom will rest 8.5 to 12.7 mm (1/3 to 1/2 in.) above the bottom of the outer crucible.

5.3 Nickel-chromium Crucible

With closely fitting cover. It can be used as alternate crucible materials. The crucible shall not be less than 10 or more than 20 ml capacity, not less than 25 or more than 35 mm in diameter, and not less than 30 or more than 35 mm in height. Nickel-chromium crucibles shall be heat-treated for 4 h at 500°C to ensure that they are completely oxidized prior to use.

5.4 Vertical Electric Tube Furnace

For coal or coke. The electric furnace shall be a vertical tube furnace. The furnace can be of the form shown in Fig. 1. It shall be regulated to maintain a temperature of $950 \pm 20^\circ\text{C}$ in the crucible, as measured by a thermocouple positioned in the furnace. The furnace shall accommodate sparking coals and meet the specifications set forth in 6.3.2.

5.5 Balance

Sensitive to 0.1 mg.

6 PROCEDURE

6.1 The sample shall be the material pulverized to No. 60 (250- μm) sieve.

6.2 Procedure for Nonspeaking Coals and Cokes

6.2.1 Weigh 1 g of the sample in a weighed platinum crucible, close with a cover which fits closely enough so that the carbon deposit from bituminous, subbituminous, and lignite coals does not burn away from the underside, place on platinum or nichrome-wire supports and insert directly into the furnace chamber, which shall be maintained at a temperature of $950 \pm 20^\circ\text{C}$, and lower immediately to the 950°C zone. Regulation of the temperature to within the prescribed limits is critical. After the more rapid discharge of volatile matter has subsided, as shown by the disappearance of the luminous flame, or in the case of coke, after 2 or 3 min, inspect the crucible (*see Note*) to verify that the lid is still properly sealed. If necessary, reseal the lid to guard against the admission of air into the crucible. Do this as rapidly as possible by raising the crucible to the top of the furnace chamber, reposition the lid to more perfectly seal the crucible, then lower the crucible immediately back to the 950°C zone. After heating for a total of exactly 7 min, remove the crucible from the furnace and without disturbing the cover, allow it to cool. Coke should be cooled in a desiccator. Weigh as soon as cold. The percentage loss of weight minus the percentage moisture equals the volatile matter. With some strongly caking low-volatile and medium-volatile bituminous coals, the coke button

can be broken with explosive violence due to the liberation of volatile matter within the button. This is usually designated as popping. Such popping can blow the lid off the crucible and cause mechanical losses of the coked material. When such popping is observed, the determination shall be rejected and the test repeated until popping does not occur.

NOTE — Inspection of the crucible can be aided by the use of a mirror held above the furnace well.

6.3 Modified Procedure for All Sparking Fuels

6.3.1 Fuels that do not cake or cake weakly when volatile matter is determined shall be watched closely for sparking during the heating period (*see Note 2*); also, at the end of the test the crucible cover shall be inspected for ash deposits, and the presence of such deposits shall be considered as evidence of sparking.

6.3.2 All fuels that spark when the volatile matter is determined by the methods described in 6.1 shall be treated as follows:

- The sample shall be given a preliminary gradual heating such that a temperature of $600 \pm 50^\circ\text{C}$ is reached in 6 min (*see Note*).
- After this preliminary heating the sample shall be heated for exactly 6 min at $950 \pm 20^\circ\text{C}$.
- If sparking is then observed, the determination shall be rejected and the test repeated until no sparking occurs either during the preliminary heating or during the 6 min period at 950°C .
- Remove the crucible from the furnace, cool on a metal cooling block, and weigh. To ensure uniformity of results, keep the cooling period constant and do not prolong beyond 15 min. The percentage loss in weight minus the percent moisture is the volatile matter. All analyses by this test method shall be so marked when reported to indicate that the modified procedure was used.

NOTE — If a tubular furnace of the Fieldner type (Fig. 1) is used for the determination of volatile matter, the preliminary gradual heating can be accomplished by moving the crucible to predetermined positions in the cooler top zone of the furnace. Due to variations in the heating characteristics of the furnace, the operator shall predetermine by thermocouple the proper positions to meet a preliminary heating rate as specified in 7.3.2. A mechanical device to lower the crucible into the furnace can be used to facilitate control of the lowering operation.

7 CALCULATION

7.1 Calculate the weight loss percent as follows:

$$\text{Weight loss, percent} = [(A - B)/A] \times 100$$

where,

A = weight of sample used, g, and

B = weight of sample after heating, g.

7.2 Calculate the volatile matter percent in the analysis samples as follows:

Volatile matter in analysis sample, percent = $C - D$

where,

C = weight loss, percent, and

D = moisture, percent.

8 PRECISION AND BIAS

Method precision shall be established based on round robin outcome.

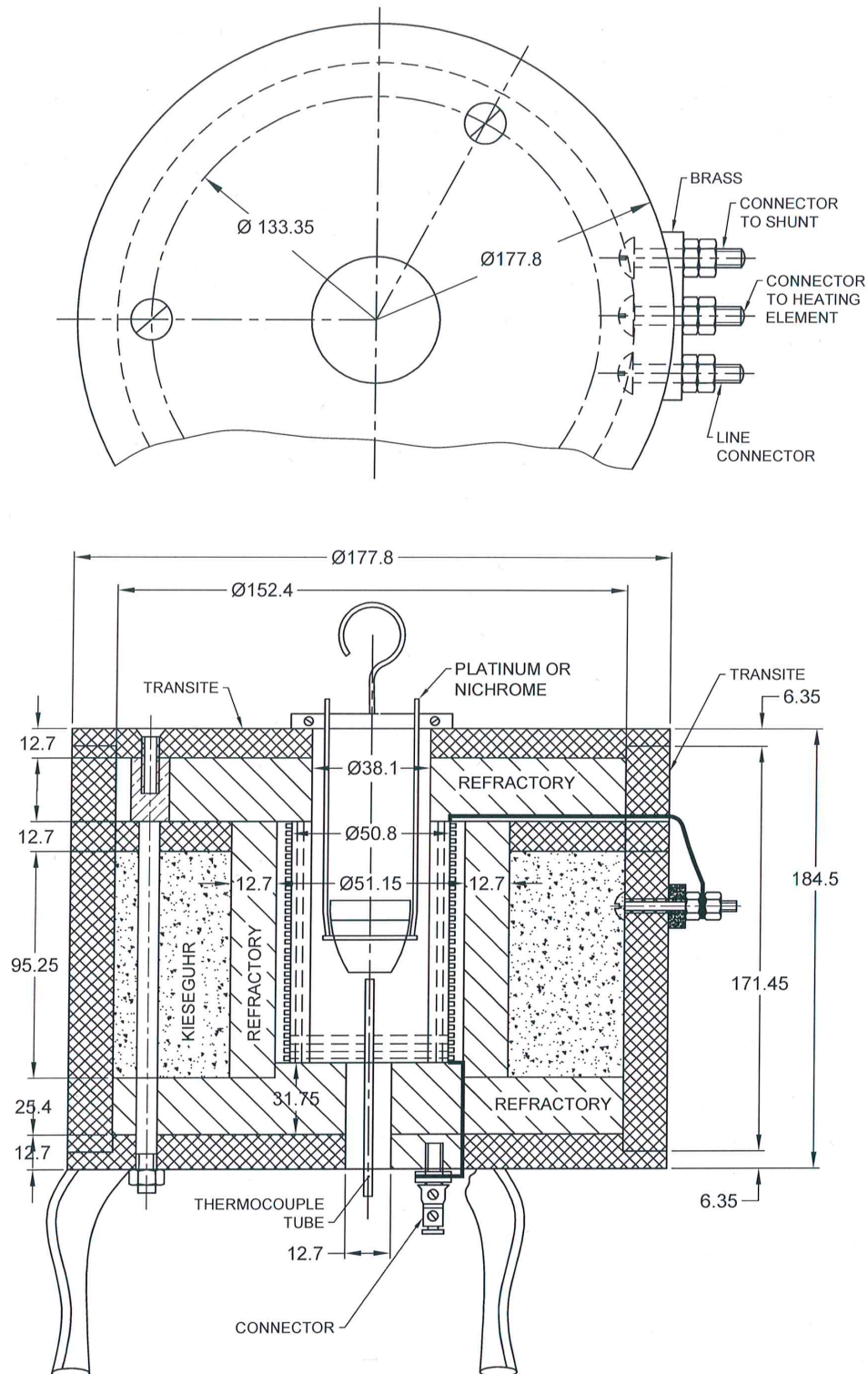


FIG. 1 ELECTRIC FURNACE FOR DETERMINING VOLATILE MATTER

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